IN THE UNITED STATES PATENT AND TRADEMARK OFFICE Attorney Docket No.: 2354-440 (FF39606/06)

Applicant(s)	Alexander et al.)
Serial No:	10/594,649) Examiner:) Nguyen, Haidung D) Art Unit:) 1796
Confirmation No:	1610	
Filed:	September 27, 2006	
For:	CERAMIFYING COMPOSITION FOR FIRE PROTECTION)))

<u>DECLARATION UNDER 37 CFR § 1.132 OF</u> <u>PULAHINGE DON DAYANANDA RODRIGO</u>

Commissioner of Patents P.O. Box 1450 Alexandria, Virginia 22313-1450

Dear Sir:

- I, Pulahinge Don Dayananda Rodrigo; hereby declare:
- 1. I am the Pulahinge Don Dayananda Rodrigo, named as an inventor in the application.
- 2. I am qualified as a Materials Scientist having obtained the degree of Doctor of Philosophy (PhD) in Materials Science from the University of Limoges, France, in 1986.
- 3. My work experience includes over 20 years in the materials industry involved in research and development of ceramic materials. Since 2002 I have been employed as a Senior Research Fellow, at Monash University, Clayton, Victoria, Australia, in which capacity I have conducted research in relation to polymeric compositions filled with inorganic materials, which, in a fire, form a ceramic so as to provide a barrier to the spread of fire and/or preserve communications cables required for evacuation, or fire fighting in the event of a fire.

A copy of my work listing is enclosed as Exhibit 1 hereto.

- 4. As a result of my qualifications and work experience, I have attained a high level of knowledge in relation to the chemical and physical properties of ceramics in general and, in particular, filled polymer compositions which form a ceramic residue in a fire.
- I have read the Office Action having a date of mailing of January 26, 2009 and the Patent specification of Vexler at al. (US 2004/0216914) and Casiraghi (US6555605).
- 6. The improvement referred to in Vexler et al. is the use in a polyolefin matrix of nano-sized particles such as nano-clay and a flame retardant additive package. The specification explains in paragraph [0017] the improvement imparted by nano-clays treated with an organic modifier.
- 7. Vexler et al. uses an intumescent system (see paragraphs 28 to 32) described generally in paragraphs [0027] and [0037]. The intumescent system contains a desiccant such as a phosphate, a carbohydrate (such as a starch or a polyhydric alcohol), a source of nitrogen and an expanding agent.
- 8. The Casiraghi invention is based on the inventors finding that the combination of magnesium hydroxide and calcium carbonate with a phosphorous-nitrogen intumescent system gives a much better result than either of the metal compounds without the other. The phosphorous-nitrogen intumescent component is similar to that of Vexler with suitable phosphorous compounds described in column 3, line 66 to column 4, line 13. Casiraghi teaches that the organic phosphorous compounds are particularly preferred and triphenyl phosphate, in combination with melamine, is the best phosphorous-nitrogen intumescent system.
- 9. I was asked by the Australian Patent Attorney for Ceram Polymerik Pty Ltd to consider Vexler et al. and Casiraghi and prepare a composition consistent with their teachings. I chose to use Composition 5 of Casiraghi as a base composition, as Vexler has little information on the amounts of components. I modified the composition of Casiraghi (as taught by Vexler) by incorporating approximately 2.3% of the composition of nano-clay having a modified surface to

improve the miscibility of particles in the polymer matrix. In accordance with a general teaching of Vexler et al., I chose the product nano-clay modified with dimethyl, dehydrogenated tallow, quaternary ammonium chloride (see Vexler et al. paragraph [0017]). Attached as Exhibit 2 are details of the product CLOISITE 2A I used as the nano-clay.

The corresponding composition of the present invention was prepared by replacing the triphenyl-phosphate and melamine combination with ammonium polyphosphate in accordance with the present invention.

To produce a corresponding composition of the present invention the inorganic fillers of the Casiraghi composition (with Vexler et al. modification) were changed in accordance with the present invention by omitting the nano-clay and replacing part of the magnesium hydroxide component and all of the calcium carbonate with talc, which is a silicate mineral in accordance with the present invention containing magnesium.

The two compositions are listed in Table 1 below:

Present Invention (parts)	Material	Vexler & Casiraghi (parts)
100	Polyolefin (EVA/PE in 80/20 ratio)	100
·	CaCO₃ (Omyacarb 2T)	75
75	Talc (Unimin T38A)	-
30	Mg(OH)₂ (Magshield SNB 10)	75
58	Ammonium polyphosphate (FR Cross 484)	-
**************************************	Nano-clay (Cloisite 20A)	6 (2.3% total composition)
	Triphenyl phosphate	5
*	Melamine	2
263	TOTAL	263

10. The compositions, shown in Table 1 above, were prepared by mixing in a Haake internal mixer and specimens of approximately 70mm x 25mm x 4mm were made by hot pressing at 170°C for 10 minutes.

- To examine the fire barrier properties of the compositions of Table 1 the 11. specimens formed in accordance with clause 12 were fired in a muffle furnace by heating to 900°C at a rate of ~ 12°C/min and held at that temperature for 30 minutes. After cooling down to room temperature, the specimens were visually examined and photographs of the compositions are reproduced in Exhibit 3. In the photographs the appearance of the "Ceram" composition of the present invention is compared with the composition of Vexler and Casiraghi under the same conditions. Referring to Figure 1, the residue from the composition of the present invention was crack-free and was relatively strong. The residue from Vexler and Casiraghi composition had visible cracks. When attempting to remove from the supporting refractory brick, the residue from Vexler and Casiraghi Example disintegrated under the mild pressure exerted by the fingers (see Figure 2). When transferred on to another surface, the residue from Vexler and Casiraghi Example turned into a pile of ceramic powder/weak agglomerates (see Figure 3).
- 12. The strength of the residues referred to in clause 11 were to be tested for flexural strength by three point bending using an Instron testing machine. The composition of Vexler and Casiraghi could not sensibly be tested as it was too fragile. The composition of the present invention had a strength of 724 kPa (0.72 MPa). This difference in strength is very significant and would provide a significant advantage in the case of structures such as cable in the case of a fire. The significantly greater strength will maintain the insulation of critical building services for a much longer period in the event of a severe fire.

I further declare that all statements made in this declaration of my own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that wilful false statements may jeopardize the validity of the application or any patent issuing therein.

Date: 30.04 - 2009

Pulahinge Don Dayananda Rodrigo

P D D Rodrigo

Pulahinge Don Dayananda RODRIGO

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WORK HISTORY/ACCOMPLISHMENTS

MONASH UNIVERSITY, AUSTRALIA

Dec. 2002 -Present

Senior Research Fellow, Department of Materials Engineering

- Conduct independent hands-on research on developing fire-resistant polymer-ceramic composite materials for various passive fire protection applications including insulation for fire-rated electric cables. Play a key role in determining the direction of the research project at all stages. This work has lead to the development of many distinct classes of ceramifying polymeric materials suitable for use in fire-rated electric cables and in other fire-barrier applications. One of these developments has already been commercialized, winning two awards (CRC Association Award for Excellence in Innovation 2004 and CRC for Polymers Chairman's Award for Excellence in Commercialization 2004). Other classes of new fire-barrier materials have been developed for use in applications such as fire-door glazing seals and fire-rated elements in building structures.
- Supervise the work of junior researchers associated with the above project.
- Coordinate the work under the above project, done by many researchers at seven different sites (Monash University, UNSW, RMIT, CSIRO, Olex Cables, Ceram Polymerik and DSTO).
- Took a leading role in identifying intellectual property resulting from the above research work, establishing clearly the intellectual property position and finalising six patent applications in consultation with patent attorneys.
- Developed a procedure for evaluating high-temperature electrical properties of ceramifying cable insulations.
- Supervised post-graduate and undergraduate researchers working in areas related to fire-resistant polymeric materials.
- Taught "Structural Ceramics" (Fourth Year Subject in Materials Engineering) and "Ceramic Processing" (Third Year Subject in Materials Engineering).

CERAMIC FUEL CELLS LIMITED, AUSTRALIA Senior Materials Scientist Sept. 2000 - Aug. 2002

• Developed alumina-reinforced yttria-stabilised zirconia (YSZ) materials and tape casting processes for manufacturing electrolyte and interconnect plates for an all-ceramic, planer, solid oxide fuel cell stacks.

Resume P D D Rodrigo

- Developed YSZ-NiO-CeO₂ composite materials and manufacturing processes for anode and fuel distribution layers of electrolyte supported solid oxide fuel cells.
- Developed a ceramic gas separator / interconnect plate with built-in current collectors and electrical connectors, capable of being thermally cycled from 850 °C to ambient temperature with no detectable performance or structural degradation.
- Designed and built equipment to measure the gas permeability of thin porous layers deposited on dense substrates.
- Developed and documented functional and manufacturing requirements of all components of an internally manifolded, all-ceramic, solid oxide fuel cell stack of electrolyte-supported, planer, cell design.
- Supervised and provided technical leadership to many taskforces responsible for the design and manufacture of solid oxide fuel cell components from concept generation stage through proof of concept stage to verification & validation stage.
- Headed the taskforces responsible for (1) developing gas separator / interconnect plate, (2) verifying performance of all-ceramic solid oxide fuel cell stacks and (3) characterising anode.
- Carried out day-to-day resource management of the Materials Processing group.

CSIRO MANUF. SCIENCE & TECH., AUSTRALIA Research Scientist July 1997 - July 2000

- Conducted an extensive study on the effects of die-casting conditions, section thickness of the casting and application of protective coatings (anodic and powder coatings) on the mechanical properties of die-cast Mg alloy (AZ91D, AM60B, AS21) components. Ensured successful completion of this project, sponsored by the Department of Energy, USA, by co-ordinating R&D activities in 7 locations (three in USA, three in Australia and one in New Zealand) while making over 20,000 die-castings using a cold chamber machine and a seven-cavity die and characterising them.
- Conducted further research on various aspects of Die-Casting Mg Alloys with emphasis on the following:
 - effects of casting conditions on the microstructural features of the products.
 - correlation between the microstructural features and mechanical properties.
 - optimum die casting conditions for common commercial Mg alloys.
 - effects of low temperature ageing on microstructure and mechanical properties of AZ91D and AM60B alloy die-castings.
 - causes of banded defects.
- Supervised a postgraduate research project and participated in the formulation of project proposals to attract external funds.

THE UNIVERSITY OF MELBOURNE, AUSTRALIA Feb. 1995 - June 1997 Research Fellow, Dept. of Mechanical and Manufacturing Engineering

 Designed and setup a facility having a 15kg/batch capacity for making ceramic particulate reinforced Metal Matrix Composites (MMCs) by semi-liquid/semi-solid processing. Resume P D D Rodrigo

• Developed a process to make aluminium MMCs with particulate reinforcements (mainly hollow flyash), optimised process parameters and transferred the technology to build a pilot plant of 100kg/batch capacity.

- Developed aluminium alloys and MMCs having globular grain structures, suitable for semi-solid forming.
- Contributed to the formulation of project proposals to attract external funds and supervised undergraduate research projects.

THE UNIVERSITY OF MORATUWA, SRI LANKA

Head/Senior Lecturer, Department of Materials Engineering, Aug. 1988 - Dec. 1994 Senior Lecturer, Department of Materials Engineering, June 1986 - Aug. 1988 Assistant Lecturer, Department of Materials Engineering, Nov. 1981 - Dec. 1982

- Taught many subjects including Properties of Materials, Strength of Materials,
 Ceramic Science, Ceramic Manufacture, Special Ceramics, Engineering Chemistry,
 Composites, Mechanical Behaviour of Materials, Microscopy, X-ray Diffraction,
 Corrosion, Non-Destructive Testing and Electrical Properties of Materials.
- Supervised 19 undergraduate research projects and 4 postgraduate research projects.
- Established a well-equipped materials characterisation laboratory consisting of facilities for XRD, SEM, Thermal Analysis (DTA, TGA, TMA, DSC), Mechanical Testing, IRS and AAS. Funds were raised by undertaking contract research and also from donor agencies.
- Prepared and implemented programs to develop academic and research strengths of the Department. This included liaising with other academic institutions and local industries.
- Nearly doubled the Materials Engineering student intake by introducing programs to increase the employability of graduates.
- Provided leadership, guidance and encouragement to the academic staff and conducted their performance planning and evaluation.
- Advised various government bodies and industries on matters related to the field of Materials Engineering.
- As a member of the University Senate, participated in university-wide policy planning and decision making with regard to both academic and administrative matters.

SRI LANKA STANDARDS INSTITUTION

Mar. 1988 - July 1994

Consultant

- Chaired the technical committee on Plastics in Engineering Applications.
- Contributed to formulation and revision of national (Sri Lanka) standards in the branches of Welding, Non-Destructive Testing, Metals in Engineering Applications and Plastics in Engineering Applications.

Resume P D D Rodrigo

<u>DANKOTUWA PORCELAIN (PVT.) LTD., SRI LANKA</u> Dec. 1988 - Dec. 1990 Director (Technical)

- Advised technical staff on matters related to Fine China tableware production by slip casting, plastic forming and extrusion.
- Participated in general policy planning and decision making as a member of the Board of Directors.
- Contributed to improving the quality and output of products and reducing the cost of production to a level that enabled the company to become a major supplier of high quality porcelain tableware to exclusive markets in USA, Japan and Europe.
- Contributed to wiping-off previously accumulated losses in 2 years, and winning the prestigious Presidential Award for export oriented companies in Sri Lanka.

THE OPEN UNIVERSITY OF SRI LANKA Oct. 1987 - Dec. 1990 Senior Consultant in Mechanical Engineering and Technology

- Formulated course structures in Mechanical Technology.
- Wrote and edited lessons in Metallurgy, Welding and Mechanical Behaviour of Materials for use in distance education program.

THE UNIVERSITY OF LIMOGES, FRANCE Graduate Research Assistant Dec. 1982 - May 1986

- Developed processes to produce dense mullite and mullite-zirconia ceramics by powder pressing and reaction sintering.
- Supervised undergraduate research projects.

CEYLON STEEL CORPORATION, SRI LANKA Sri Lanka's largest manufacturer of steel products Assistant Foundry Engineer (Traince) July 1980 - Sept. 1980

- Developed a mould for testing fluidity of molten brass.
- Contributed to alloy/product development and supervision of the sand casting department.

CEYLON CERAMICS CORPORATION, SRI LANKA, June 1979 - Aug. 1979 Sri Lanka's largest manufacturer of ceramic sanitaryware, tableware and mosaic tiles. Assistant Production Engineer (Trainee)

- Improved the crazing resistance of tableware by re-formulating the glaze and the body and modifying the firing cycle.
- Contributed to glaze development and supervision of the forming department.

Resume P. D. D. Redrigo

EDUCATIONAL QUALIFICATIONS

- PhD (Docteur de l'Universite) in Materials Science Higher National School of Industrial Ceramics (ENSCI)/University of Limoges, France (1983 1986).
- **DEA** (Equivalent to MSc) in Materials Science University of Limoges, France (1983 1984).
- BSc in Materials Eng. (First Class Honours) University of Moratuwa, Sri Lanka (1977 1981).

TRAINING

- Working Collaboratively within a Research Project Team Environment (one-day course) School of Enterprise, The University of Melbourne, 28 Sept. 2007.
- Intellectual Property Workshop Innovation Law, Philip Mendes & Bradley Thomas, Brisbane, 27 Sept. 2007.
- Leadership and Innovation Course with Commercialisation Bootcamp Centre for R&D Leadership, The University of Melbourne and Australian Institute for Commercialisation, Brisbane 14 Aug. to 18 Aug. 2006.
- Multi Factor Solutions for Product & Process Problems Design of Experiments, RMIT University, May 2001.
- Applied Aspects of Neutron Scattering Bhabha Atomic Research Centre, Bombay, India 21 Nov. to 10 Dec. 1993.
- Materials Characterisation Using Low and Medium Neutron Flux Reactors China Institute for Atomic Energy, Beijing, China 26 Oct. to 13 Nov. 1992.
- NDT (Ultrasonic Techniques) Atomic Energy Authority, Sri Lanka 24 July to 9 Aug. and from 22 Sept. to 8 Oct. 1992.

AWARDS RECEIVED

Team awards

- BHERT (Business/High Education Round Table Awards for Outstanding Achievement in Collaboration in Research & Development and Education & Training) Award for Best Collaboration Involving a CRC 2004.
- CRC Association Award for Excellence in Innovation 2004.
- CRC for Polymers Chairman's Award for Excellence in Commercialisation 2004.

Individual awards

- CFCL Award for Best Performing Technical Taskforce Leader 2001.
- Commonwealth Academic Staff Fellowship 1994.
- French Government Postgraduate Award 1982.
- Union Carbide Ceylon Ltd. Prize for the best performance at the B. Sc. Eng. Fourth Year Examination in Materials Engineering, University of Moratuwa 1981.
- Ceylon Ceramics Corporation Scholarship for the best performance at the B. Sc. Eng. Third Year Examination in Materials Engineering, University of Moratuwa 1980.
- University of Moratuwa Scholarship for the best performance at the B. Sc. Eng. First Year Examination in Materials, Chemical & Mining Engineering, University of Moratuwa – 1978.

SELECTED PUBLICATIONS

- 1. P.D.D.Rodrigo and P.Boch, "High Purity Mullite Ceramics by Reaction Sintering", Int. J. High Technol. Ceram., 1 (1985) pp. 3-30.
- 2. P.D.D.Rodrigo and P.Boch, "Preparation of High Purity Mullite Ceramics", Science of Ceramics 13, Editions de Physique Publ., Paris, 1986, J. Phys. 47, pp. Cl-411.
- 3. P.D.D.Rodrigo and P.Boch, "Effects of Al₂O₃: SiO₂ Ratio of the Starting Mixture on the Composition, Microstructure and Mechanical Properties of Mullite Produced by Reaction Sintering", Transactions of the Materials Research Society of Japan, Elsevier Science Publishers Ltd., UK, London (1990) pp.180-191.
- 4. B.P.S.Peiris, P.G.R.Dharmaratne and P.D.D.Rodrigo, "Improvement of the quality of semi-precious gem stones by heat treatments", Report to Sri Lanka Gem Corporation, Colombo, Sri Lanka, June 1992.
- L.J.P.Jayantha Manel, H.S.Amarasekara and P.D.D.Rodrigo, "Anatomical Structure, Mechanical Properties and Preservation of Bamboo", Report to CISIR, Colombo, Sri Lanka, September 1994.
- 6. P.D.D.Rodrigo, N.Setargew, P.Fitzgerald, G.Withers and K.Xia, "Solidification Processing of Aluminium Casting Alloy Reinforced with Ceramic Microspheres for Thixoforming", *Proceedings of 126th TMS Annual Meeting*, Orlando, Florida, USA, February 1997, Light Metals 1997, pp. 953-960.
- 7. P.D.D.Rodrigo, M.Murray, H.Mao, J.Brevick, C.Mobley, V.Chandrasekar, and R.Esdaile, "Effects of Section Size and Microstructural Features on the Mechanical Properties of Die Cast AZ91D and AM60B Magnesium Alloy Test Bars", SAE International Congress and Exposition, Detroit, Michigan, USA, March 1999 Technical Paper No. 1999-01-0927.
- 8. P.D.D.Rodrigo, M. Murray, H. Mao, V. Chandrasekar, Y. Kisioglu, A. Deshpande, J. Brevick, C. Mobley, and R. Esdaile, "Fatigue Properties of Die Cast Magnesium Alloys", SAE International Congress and Exposition, Detroit, Michigan, USA, March 2000 Technical Paper No. 00M-169.
- 9. P.D.D.Rodrigo and V. Ahuja, "Effect of Casting Parameters on the Formation of 'Pore-Bands' in Magnesium Die-Castings", Proceedings of the Second Israeli International Conference on Magnesium Science & Technology, Dead Sea, Israel, February 2000, pp. 97-104.
- 10.P.D.Rodrigo and M. T. Murray, "The Influence of Coatings on Mechanical Properties of Die-Cast Magnesium Alloys", ADCA Bulletin, 87 (2001) pp. 12-20.
- 11. Olivier Bellon, Raj Ratnaraj and **Don Rodrigo**, "10YSZ based electrolyte materials for electrolyte supported SOFCs", *Proceedings of 5th European Solid Oxide Fuel Cell Forum*, Lucerne, Switzerland, July 2002, pp. 184-190.

12.K.W. Thomson, **P.D.D. Rodrigo**, C. M. Preston and G.J.Griffin, "Ceramifying Polymers for Advanced Fire Protection Coatings" European Coatings Journal, 12 (2006), pp. 34-39.

PATENTS (Co-author)

- 1. International Patent Application No. PCT/AU02/00939, "A fuel cell gas separator plate" (International publication number WO 03/007403).
- 2. International Patent Application No. PCT/AU2003/000968, "Fire-resistant silicone polymer compositions" (International publication number WO 2004013255).
- 3. International Patent Application No. PCT/AU2003/001383, "Fire resistant polymeric compositions" (International publication number WO 2004035711).
- 4. International Patent Application No. PCT/AU2004/000410, "Cable and article design for fire performance" (International publication number WO 2004088676).
- 5. International Patent Application No. PCT/AU2005/000465, "Ceramifying composition for fire protection" (International publication number WO 2005095545).
- 6. Australian provisional patent application, "Polymer foam and foam articles for fire protection" (No. 2007902417).
- 7. Australian provisional patent application, "Polymer foam containing heat expandable material and foam articles for fire protection" (No. 2007902415).

Southern Clay Products Cloisite® 20A Nanoclay

Categories: Other Engineering Material; Additive/Filler for Polymer; Polymer

Material Cloisite® 20A is an additive for plastics to improve various plastic **Notes:** physical properties, such as reinforcement, HDT, CLTE and barrier.

Information provided by Southern Clay Products

Key Words: Quaternary ammonium salt modified natural montmorillonite polymer

additive.

Vendors: No vendors are listed for this material. Please click here if you are a

supplier and would like information on how to add your listing to this

material.

Download to Excel (requires Excel and Windows)

Export data to your CAD/FEA program

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Physical Properties	Metric	English	Comments
Specific Gravity	1.77 g/cc	0.0639	
		lb/in³	
Bulk Density	<u>0.118</u> g/cc	0.00425	Loose
		lb/in³	
	<u>0.2171</u> g/cc	0.007841	Packed
		lb/in³	
Loss On Ignition	38.0 %	38.0 %	
Particle Size	<= <u>2.00</u> μm	<= <u>2.00</u>	10%
		μm	
	<= <u>6.00</u> μm	$\leq \underline{6.00}$	50%
		μm	
	<= <u>13.0</u> μm	<= <u>13.0</u>	90%
		μm	

Mechanical Properties	Metric	English	Comments
Hardness, Shore D	83	83	5% Cloisite®
			reinforced Nylon 6
Tensile Strength,	<u>101</u> MPa	<u>14600</u> psi	5% Cloisite®
Ultimate			reinforced Nylon 6
Elongation at Break	8.00 %	8.00%	5% Cloisite®
			reinforced Nylon 6
Modulus of Elasticity	<u>4.657</u> GPa	<u>675.5</u> ksi	5% Cloisite®
			reinforced Nylon 6
Flexural Modulus	3.78 GPa	<u>548</u> ksi	5% Cloisite®
			reinforced Nylon 6
Izod Impact, Notched	<u>0.270</u> J/cm	<u>0.506</u> ft-	5% Cloisite®

lb/in

reinforced Nylon 6

Thermal Properties

Metric

English

Comments

Deflection Temperature

96.0 °C

205 °F

5% Cloisite®

at 0.46 MPa (66 psi)

20.0

<u>05</u> °F

reinforced Nylon 6

Processing Properties

Metric

English

Comments

Moisture Content

<= 2.00 %

<= 2.00 %

Descriptive Properties

Modifier Concentration, meq/ 100g clay

95

Organic Modifier

dimethyl, dihydrogenated

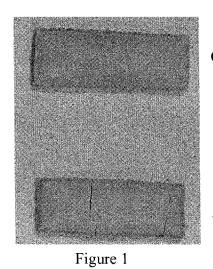
tallow, quaternary ammonium

X-Ray Diffraction d-Spacing (001)

31.5 Angstroms

Some of the values displayed above may have been converted from their original units and/or rounded in order to display the information in a consistant format. Users requiring more precise data for scientific or engineering calculations can click on the property value to see the original value as well as raw conversions to equivalent units. We advise that you only use the original value or one of its raw conversions in your calculations to minimize rounding error. We also ask that you refer to MatWeb's disclaimer and terms of use regarding this information. Click begt to view all the property values for this datasheet as they were originally entered into MatWeb.

EXHIBIT 3



Ceram

Vexler + Casiraghi

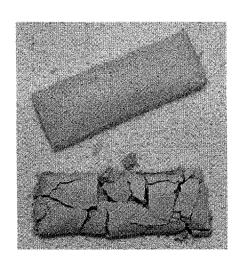


Figure 2

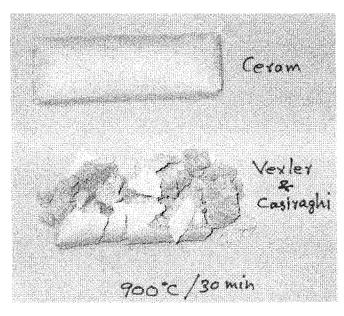


Figure 3